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REACTION BONDED SILICON NITRIDE
PREPARED FROM WET ATTRITION-
MILLED SILICON

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REACTION BONDED SILICON NITRIDE PREPARED FROM WET
ATTRITION-MILLED SILICON

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ABSTRACT

Silicon powder wet milled in heptane was dried, compacted into test bar shape, helium-sintered, and then reaction bonded in nitrogen-4 volume percent hydrogen. As nitrided bend strengths averaged approximately 290 MPa at both room temperature and 1400° C. Fracture initiation appeared to be associated with sub-surface flaws in high strength specimens and both sub-surface and surface flaws in low strength specimens.

INTRODUCTION

Previous work has shown that the dry attrition milling technique can be used to prepare a high surface area, generally fine silicon powder for use in the preparation of reaction bonded silicon nitride (RBSN) (Refs. 1 and 2). Fine particle size dry milled powder nitrided more completely than as-received coarser powder, and after nitriding it exhibited higher bend strength at both room temperature and 1400° C. However, the dry attrition-milling process as reported in Refs. 1 and 2 produces a powder with the undesirable characteristic of retaining a few large isolated silicon particles some as large as 40 μ m. Because agglomerates are avoided during wet milling, wet attrition milling more efficiently comminutes the large particles normally present in any starting powder. Since the final RBSN product reflects the initial particle size distribution, the wet milling process offers the potential for producing a more uniform structure in the product RBSN.

The objective of this study was to determine the effect of wet attrition milling of silicon powder on the properties of RBSN.

A commercial grade of silicon powder was wet attrition milled in heptane. The resultant fine powder slurry was dried, formed into test bars, and nitrided in nitrogen plus 4 volume percent hydrogen. Evaluation of the resultant RBSN (referred to as HG 103) specimens included

room temperature and 1400° C bend tests, chemistry, metallography, X-ray diffraction, and fractography. Comparisons were made to dry attrition milled silicon powder, RBSN produced from that powder, and to a commercial RBSN (Norton's NC 350).

EXPERIMENTAL PROCEDURE

Fine powder was prepared by milling as-received minus 325 mesh (44 μm) silicon powder in a nickel attrition mill using 3.2 mm diameter type 440C stainless steel balls as media and normal heptane as the milling fluid. The attrition mill is shown schematically in Fig. 1. Milling was done in air and the media to powder weight ratio was 40:1. Milling time was 7 hours. Four separate milling runs were carried out with powder charges of 60 grams of silicon in 250 ml of heptane. The resultant slurries were separated from the ball charge, mixed together, and then air dried to produce a friable powder cake. The powder cake was broken up by passing it through a 70 mesh (212 μm) sieve to facilitate preparation of test bars. The dried and sieved powder was air handleable in contrast to dry attrition milled powder which was pyrophoric in nature. Both the as-received and wet milled powder samples were analyzed for trace metallic impurity content by spectrographic analysis. Oxygen, carbon, nitrogen, and iron analyses were obtained by means of standard analytical procedures (see Table I). The specific surface area of the as-received and wet milled powder was determined by the 3-point BET technique.

Test bars were made from the milled powder by cold compacting approximately 3.2 grams of powder in a uniaxial single acting steel die without binder at a pressure of 70 MPa. It was observed (qualitatively) that the green strength of bars pressed from silicon powder ground in heptane was less than that of bars pressed from dry ground powder, but considerably greater than that of bars prepared from as-received powder. The cold pressed bars, 10 mm wide by 3 mm thick by 76 mm long, were subsequently isostatically pressed at 350 MPa. A specimen of as-received powder was prepared for SEM examination by direct isostatic compaction at 350 MPa. Representative pressed samples prepared from the as-received and from the wet milled silicon powder were fractured and examined by scanning electron

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microscopy (SEM). The green density of representative samples prepared from the milled silicon powder was determined by the vacuum mercury displacement technique.

Test bars prepared from the wet milled silicon powder were weighed and stacked in an open square array. The bottom bars rested on RBSN setters placed on a flat high-purity alumina tray. Twenty 3.2 gram silicon bars constituted a furnace load. Specimens were located within the central 15 cm of the furnace hot zone where the temperature variation could be held to $\pm 2^{\circ}$ C. The furnace tube was high purity alumina and heat was provided by external silicon carbide heating elements. The furnace tube was sealed, leak checked and then a flow of high-purity helium of 500 ml/minute was established. After allowing 2 hours for purging, the temperature was increased linearly over a 4 hour period to 1150° C. After 4 hours of sintering at 1150° C the power was turned off and the samples cooled to ambient temperature.

At the completion of the sintering cycle, the helium flow was stopped, and a nitrogen-4 volume percent hydrogen flow of 0.10 ml/minute was established. The nitriding atmosphere flow was controlled with a precision 1° taper needle valve and calibrated with a bubble flow meter. Both the nitrogen and hydrogen were purified prior to introduction into the furnace. The oxygen and moisture contents of both gases were of the order of 1-2 ppm. Exit gas from the nitriding furnace passed through an oil filled bubbler which established a backpressure of 2.3 MPa.

A nitriding cycle developed by Mangles (Ref. 3) having a maximum temperature of 1400° C was used. The cycle is shown in Fig. 2 along with the cycle used in the previous studies of RBSN prepared from dry attrition milled silicon reported in Refs. 1 and 2. Unpublished work by the authors has demonstrated that the cycle used in this study is effective for complete nitridation of the as-received minus 325 mesh silicon powder and both dry and wet milled silicon powders, and results in the same properties in RBSN prepared from dry milled silicon as reported in Ref. 2.

After completion of the nitridation step the samples were weighed to determine weight gain, and their densities were determined by vacuum mercury

displacement. Four point bend strengths of specimens with as-nitrided surfaces were determined at room temperature and 1400° C. Broken test bars were examined at low magnification. Additional examination included light microscopy of polished cross-sections and SEM of fracture surfaces. Representative samples were also examined by X-ray diffraction. Selected peak intensities of the phases detected were analyzed by the technique developed by Gazzara and Messier (Ref. 4) to determine the amount of $\alpha\text{Si}_3\text{N}_4$, $\beta\text{Si}_3\text{N}_4$, and residual silicon present.

RESULTS AND DISCUSSION

Powder Characteristics

Wet attrition milling was very effective in reducing both the average size and the maximum size of the silicon particles present in the starting minus 325 mesh powder. Figure 3 shows the effect of 7 hours of milling on the morphology of the silicon powder as observed by SEM of fractured compacted (unsintered) specimens. As-received powder contained numerous small particles as-well-as a number of large angular particles some of which were greater than 44 μm in major dimension. No particles greater than 5 μm were observed in the milled powder. Most of the particles appeared to be one μm or less in size. In contrast, silicon powder prepared by dry attrition milling (Refs. 1 and 2) was characterized by the presence of large silicon particles (up to 40 μm)- even after milling for 18 hours. The surface area of silicon powder wet milled for 7 hours was 15.9 m^2/g , 33 times greater than the initial 0.48 m^2/g . The surface area of 15.9 m^2/g corresponds to an particle size well below 0.5 μm . By comparison, a surface area of approximately 18 m^2/g (interpolated) would be attained in 7 hours of dry milling.

The oxygen, carbon, nitrogen, and iron contents as-well-as the spectrographically determined trace metallic impurity analyses of both the as-received and milled powders are shown in Table I. Oxygen, carbon, nitrogen, and iron all increased as a result of milling. Carbon pick up is associated with the heptane used as a milling fluid. Oxygen and nitrogen were picked-up from air exposure during and after milling. Iron was introduced from wear of the grinding balls. And nickel was introduced from wear of the nickel attrition milling pot.

Compactability

Wet milled silicon was more difficult to compact than the dry attrition milled powder described previously (Refs. 1 and 2). Presumably this difference in compactability was due to the difference in the nature of agglomerates of the two powders. Dry milled powder is characterized by numerous highly irregular dense agglomerates of very fine particles. These agglomerates are difficult to break apart; they resist being forced through a screen and stay together even while being subjected to shearing forces--as when being mixed with epoxy for metallographic examination. Silicon powder does not agglomerate noticeably during wet milling. Weak agglomerates do form when the slurries are dried. They can be readily forced through a screen; and they can be sheared apart while mixing in epoxy. The authors feel that during compaction dry milled Si powder behaves as strong irregular agglomerates which can interlock while wet milled powder behaves more nearly like equiaxed individual particles. This difference in agglomerate behavior also leads to a lower compacted density for the wet milled powder-- 1.39 g/cm^3 as compared with 1.6 to 1.75 g/cm^3 for the dry milled powder (Ref. 2).

Nitridation

Examination of the nitrided bars revealed an average weight gain of slightly greater than 59 percent. The resultant density was 2.39 g/cm^3 . If there were no weight loss of silicon during sintering and nitriding the theoretical weight gain for complete nitridation should be 66 percent. As pointed out by Baumgartner (Ref. 5) however, when weight loss which occurs during the sintering step is taken into account typical weight gains for complete nitridation should be of the order of 62 percent. X-ray analysis of the nitrided bars revealed only the presence of α and $\beta\text{Si}_3\text{N}_4$ with an α/β ratio of 6. (The α/β ratio of NC 350 was 9.) No residual silicon was detected by X-ray diffraction nor was any detected in the metallographic examination which will be described later. It must be concluded, therefore, that the difference between the observed 59 percent weight gain and the predicted weight gain of 62 percent was due to the presence of SiO_2 within the Si samples.

Metallographic Examination

Several of the nitrided bars were sectioned and polished for metallographic examination. Figure 4 shows the typical structure that was observed. The polished section of HG 103 shows a complete absence of unreacted silicon and fine porosity with an occasional isolated large pore. Commercial RBSN-Norton's NC 350 also shows no unreacted silicon and a considerable number of smaller pores.

Bend Strength

Room temperature bend strength of the nitrided bars was determined by 1/3-point loading/four-point bending. The 1400° C tests utilized 1/4 point loading-four point bending. Both room temperature and 1400° C strength data are shown in Fig. 5. Twelve specimens were tested at room temperature with an average strength of 288.8 ± 47 MPa. Eight specimens were tested at 1400° C with an average strength of 286.0 ± 48 MPa. For comparison, Fig. 5 also shows the strength data for the strongest RBSN prepared from dry attrition milled silicon from Ref. 2 and for commercial RBSN (Norton's NC 350). It is obvious that the wet material is superior in strength to the dry milled material. This strength improvement occurred despite the fact that its density, 2.39 g/cm^3 , was lower than the 2.51 to 2.58 g/cm^3 densities reported in Ref. 2 for dry milled material. The density value of 2.39 g/cm^3 was, however, very similar to the measured value of 2.40 determined in this study for NC 350. The improved structural uniformity and apparent absence of any large residual silicon particles in the nitrided structure seem to be the primary factors contributing to the strength improvement in the wet milled material.

The reason for the strength superiority of commercially prepared RBSN was not evident in the optical metallographic analysis; however, strengths determined at both room temperature and 1400° C indicate a decided advantage. This occurred despite the relative parity of measured density of wet milled RBSN and NC 350. The reason for NC 350's higher strength is believed to be related to the smaller flaw size as discussed below.

Fractography

As is typical of relatively high strength RBSN, all of the bars, both experimental and commercial, in this study fractured into many pieces, including small chips from the area near the fracture origin. In most cases, however, it was possible to locate the primary fracture origin on one piece.

Macroscopically, fracture surfaces of bars of HG 103 and NC 350 appeared very much the same: dense-looking and rather shiny dark gray as opposed to the dull, grainy appearance of weak RBSN observed in a previous study (Ref. 2). Most of the fracture origins were slightly sub-surface and located near the center of the bend specimen tensile face.

SEM examination revealed some differences between the two materials. As can be seen in Fig. 6, both HG 103 and NC 350 contain a considerable amount of fine porosity. Generally the pores were much less than 1 μm across; a few however were as large as 2 μm . The porosity in the sample of HG 103 (Fig. 6(a)) appears to be more uniformly distributed than in NC 350 (Fig. 6(b)). Based on the smoothness of the fracture surface, the NC 350 seems to have fractured in a predominately transgranular mode. The HG 103, on the other hand, appears rough, which may be indicative of a partially intergranular failure..

The most important difference in the two materials is the size of the failure initiating flaws. As indicated in Fig. 7, the flaws in NC 350 tested at room temperature were generally pores 12-15 μm in size. Fracture initiating flaws in HG 103 were generally much larger, often 25-30 μm . (In fractographic terminology, "flaw size" refers to either the radius of a circular flaw or the semiminor axis of an elliptical flaw. Thus, the pores in NC 350 had diameters of 25-30 μm and the flaws in HG 103 were actually often 100-200 μm long and 45-60 μm wide with the long dimension roughly parallel to the tensile surface.)

The large flaws in the HG 103 had a somewhat "spongy" appearance as shown in Fig. 8. They were made up of a mass of material that was poorly bonded to the surrounding matrix and was seen as either a ridge or a trough depending on which half of the fracture was observed. Energy dispersive

X-ray analysis did not indicate the presence of any metallic impurities associated with the defects.

The source of the fracture initiating flaws has not yet been positively identified. However, some of them may have resulted from silicon agglomerates which resisted compaction during fabrication. This is possible because, while the silicon does not agglomerate during wet milling, the fine particles tightly agglomerate during drying. Some of these agglomerates could survive after sieving of the cake and pressing of the bars.

In both materials, bars tested at 1400° C had much the same types of flaws as at room temperature. At the higher temperature, all of the failure initiating flaws were sub-surface. Again, the flaws in HG 103 were much larger than those in NC 350.

Mecholsky, Freiman, and Rice have examined elliptical flaws in glass samples (Ref. 6). They have shown that as in the case of round flaws, there is a correlation between elliptical flaw dimensions and the observed fracture strength. They have also shown the same considerations pertinent for a wide variety of ceramic materials (Ref. 7).

Calculations of expected sizes of the failure initiating flaws based on observed bar strengths are in good agreement with actual flaw sizes in most cases. Results of these calculations are summarized in Table II. The equations discussed by Mecholsky, et. al, (Ref. 6) were used.

$$c = \frac{\phi^2 K_{Ic}^2}{\pi \sigma^2} \quad (1a)$$

$$\phi = \int_0^{\pi/2} \left(\cos^2 \theta + \frac{a^2}{b^2} \sin^2 \theta \right)^{1/2} d\theta \quad (1b)$$

where c is the critical flaw size, that is, the semiminor axis of an elliptical flaw (the smaller of a and b , defined below); ϕ is an elliptical integral of the second kind and is a function of a/b , K_{Ic} is the critical stress intensity factor (~ 2 for RBSN (Ref. 8)), σ is the fracture stress, $2a$ is the dimension of the flaw perpendicular to the

tensile surface, and $2b$ is the dimension parallel to the tensile surface (see Fig. 9). When the defect is at the surface of the bar, the right side of Eq. (1a) must be divided by the geometrical correction factor of 1.2.

Fifty percent of the predicted flaw sizes were within ± 25 percent of the actual flaw size. This range can be considered acceptable allowing for the difficulties in measuring flaw size and the approximate values of ϕ and K_{IC} used in making the calculations.

About 25 percent of the measured fracture initiating flaws were smaller than those calculated. The slow crosshead speed ($0.008 \text{ cm-min}^{-1}$) during the MOR test may have allowed slow crack growth to occur (Ref. 8) before failure so that the actual crack was larger at fracture than the measured defect. These flaws were either small pores or small inclusions.

The remaining 25 percent of the failures were from flaws larger than would be expected from the fracture strengths or predicted by Eq. (1a). These were generally very elongated flaws approximately parallel to the tensile surface of the bar. Apparently with flaws so large, failure initiates at only one portion of the defect and then propagates through the rest of the flaw which is weaker than the surrounding material.

These calculations indicate that HG 103 is inherently similar to NC 350, but its strength is reduced by the presence of very large flaws.

SUMMARY OF RESULTS

Wet attrition milling was applied as a means of eliminating large, difficult to nitride silicon particles present in silicon powder used for making experimental reaction bonded silicon nitride. The following observations were made:

1. Wet attrition milling in heptane resulted in silicon powder of fine (submicron) particle size.
2. The powder was contaminated with almost 3 percent oxygen and, to a smaller extent, by carbon, iron, and nickel.
3. The density of RBSN produced from wet milled powder was 2.39 g/cm^3 . The weight gain during nitridation was 59 percent. Phases detected by X-ray diffraction were α and $\beta\text{Si}_3\text{N}_4$ in the ratio of 6:1. No residual Si was detected.

4. Bend strengths averaged approximately 290 MPa at both room temperature and 1400° C.

5. Although residual Si particles were eliminated as defects, fracture initiating flaws in the experimental RBSN were quite large, typically 45-60 μm across. Measured bend strengths correlated with flaw size.

6. RBSN prepared from wet milled Si in this study was stronger than RBSN prepared from dry milled powder. It was, however, weaker at both room temperature and at 1400° C than commercial RBSN (NC 350).

7. The potential of wet milling for production of RBSN is evidenced by the improved strength in comparison to dry milled (MOR at 1400° C: 290 MPa vs. 220 MPa for dry milled). A further indication of the potential of the process was the increased general uniformity of HG 103 compared with NC 350. If the remaining flaws in the wet milled product can be eliminated a product superior to current commercial practice should result.

ACKNOWLEDGMENT

The writers thank S. W. Freiman for helpful discussions on the fractographic analysis.

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TABLE I. - CHEMICAL ANALYSIS OF Si, AS-RECEIVED, AFTER MILLING, AND AFTER NITRIDATION

	Weight percent												
	O ₂	C	N ₂	Fe	Al	Ca	Cr	Mg	Mn	Na	Ni	Ti	V
As-received	0.1	0.07	0.003	0.38	0.095	0.016	0.016	0.001	0.014	0.011	0.053	0.021	0.084
As-milled	2.8	.67	.016	.63	.084	.012	.012	.001	.007	^a ND	.3	.024	.1
As-nitrided	1.9	.37	-----	.47	-----	-----	-----	-----	-----	-----	-----	-----	-----

^aND = not detected.

TABLE II. - MEASURED AND CALCULATED FLAW SIZES

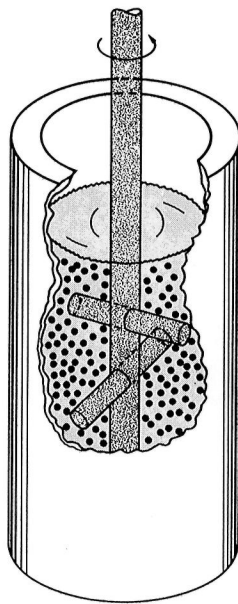
Specimen	Test temperature	Measured flaw size, μm^1		Flaw location surface or interior	Strength, MPa	Calculated flaw size, μm^1
		a	b			c
HG 103 ²	RT ↓ 1400° C ↓	30	90	I	280	20
1A		25	20	I	282	32
7		23	55	I	253	26
13		40	150	I	289	18
19		30	25	S	264	31
101A		30	30	I	295	36
101B		30	25	S	343	18
102A		35	63	I	326	20
4		18	10	I	310	22
8		25	58	I	236	30
10		20	45	I	247	28
12		40	90	I	227	33
14		18	23	I	318	25
18		20	100	I	265	20
20						
NC 350 ³	↓ RT ↓ 1400° C ↓ 1400° C ↓ 1400° C	10	15	S	265	11
2A		25	12	S	350	16
3A		20	30	I	387	15
4A		18	10	I	370	15
5A		33	25	I	340	22
1B		20	30	I	312	23
3B		15	25	I	402	13
6B						

¹a and b are the minor and major semiaxes of the measured flaw.

c is compared to the smaller of a and b.

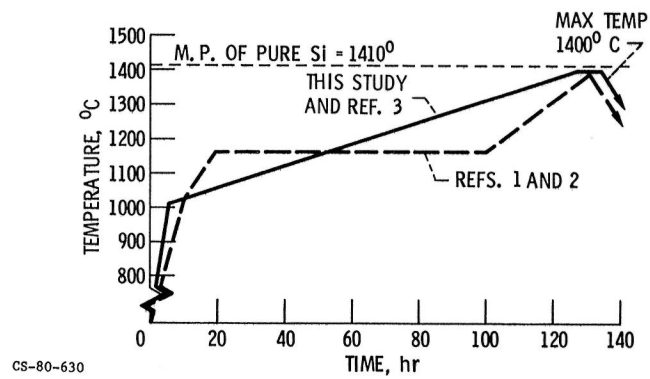
²HG 103 is RBSN prepared from wet milled silicon powder.

³NC 350 is commercially produced RBSN, Norton Company, Worcester, Mass.



CS-80-563

Figure 1. - Heptane filled attrition mill.

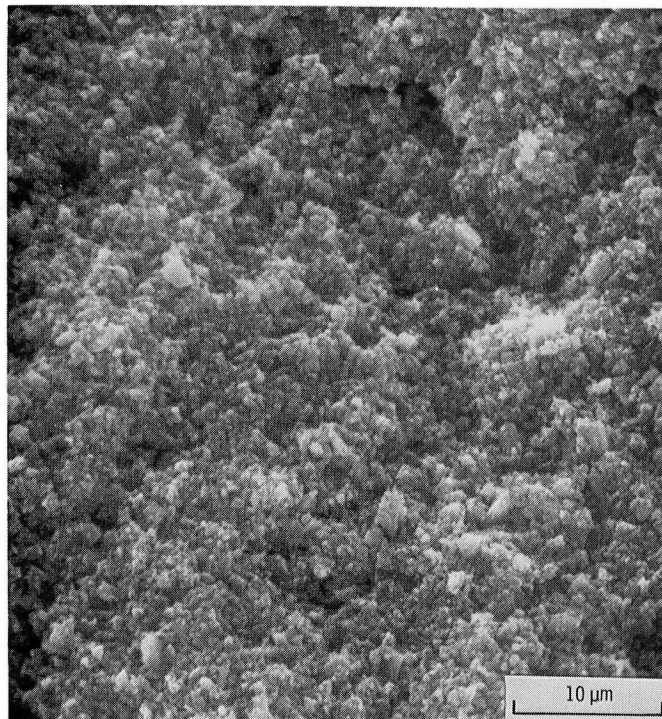


CS-80-630

Figure 2. - Nitriding cycle (ref. 3).

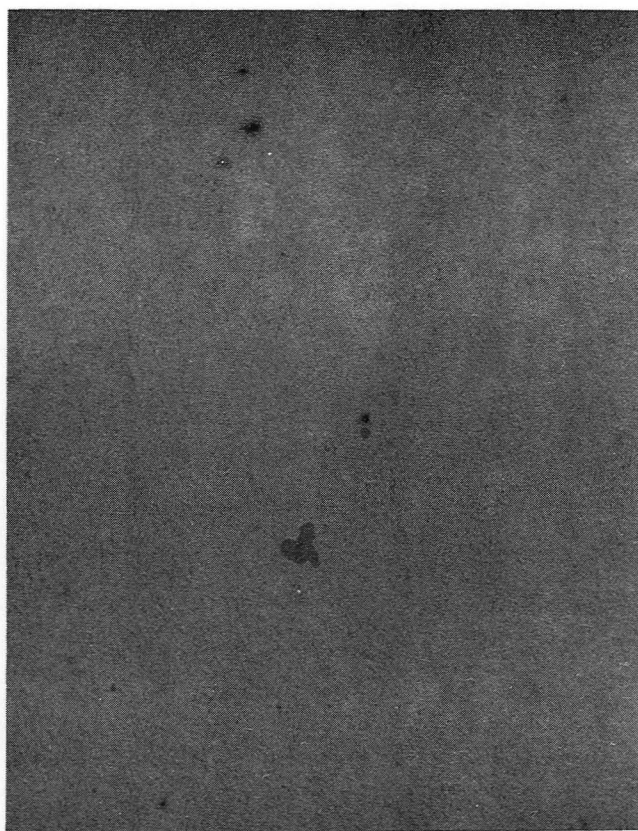


AS RECEIVED



WET ATTRITION-MILLED 7h

Figure 3. - Scanning electron micrographs of as received and wet attrition milled silicon powder.



(a) EXPERIMENTAL RBSN



(b) NC-350.

Figure 4. - Light metallographic appearance of RBSN prepared from wet milled silicon and NC-350.

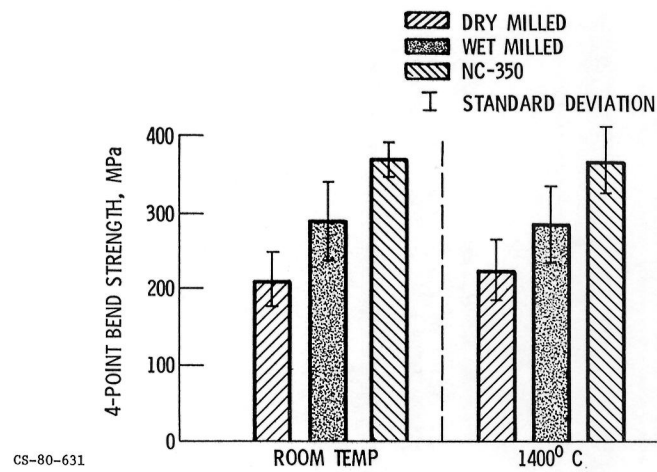
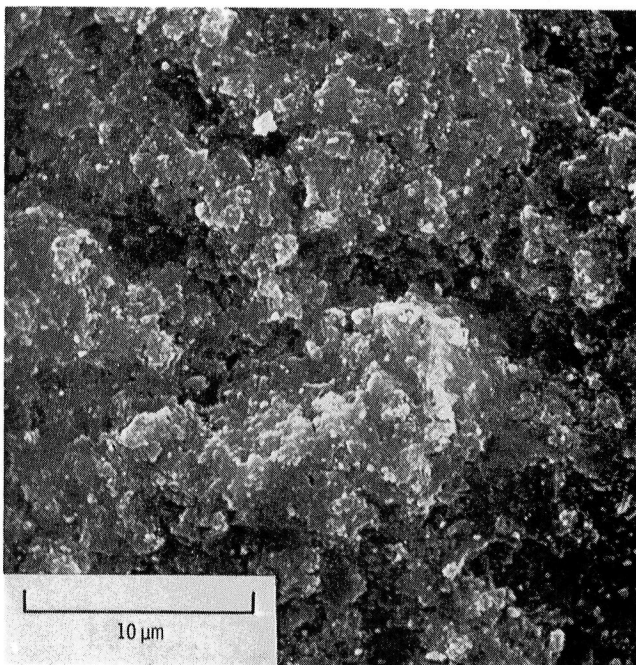
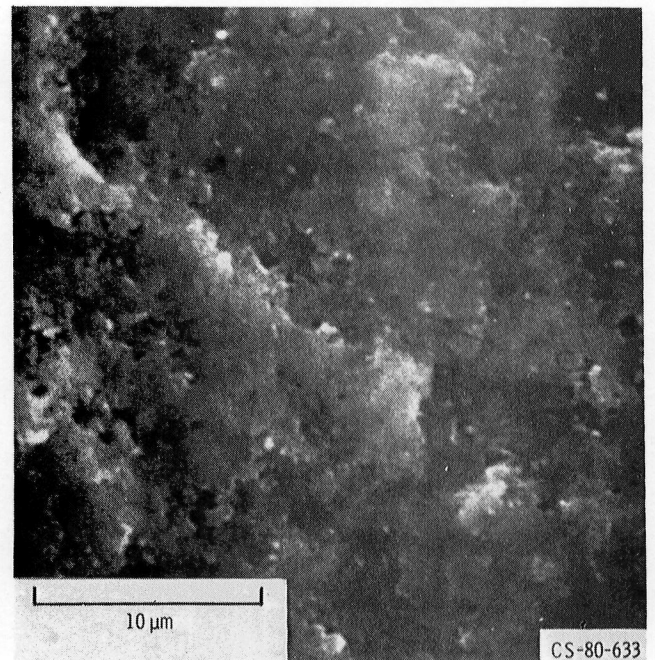


Figure 5. - Strength of reaction bonded Si_3N_4 .

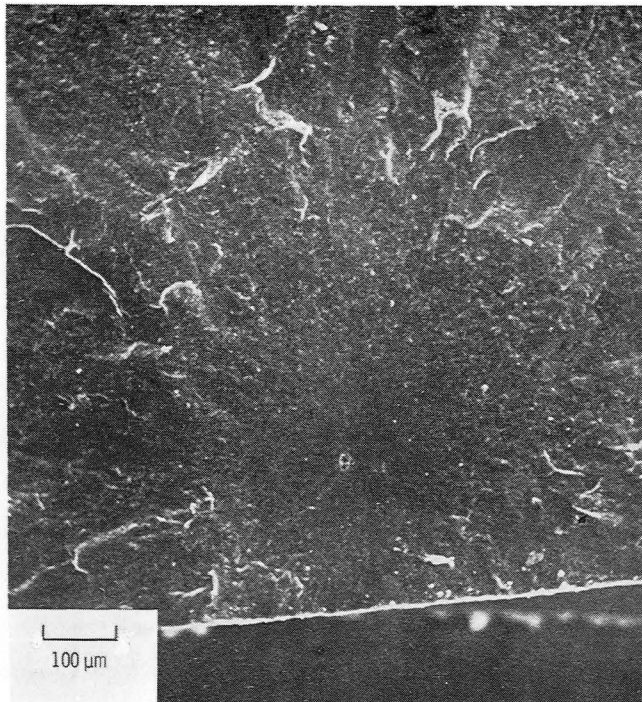


(a) BAR FROM WET ATTRITOR MILLED Si POWDER.

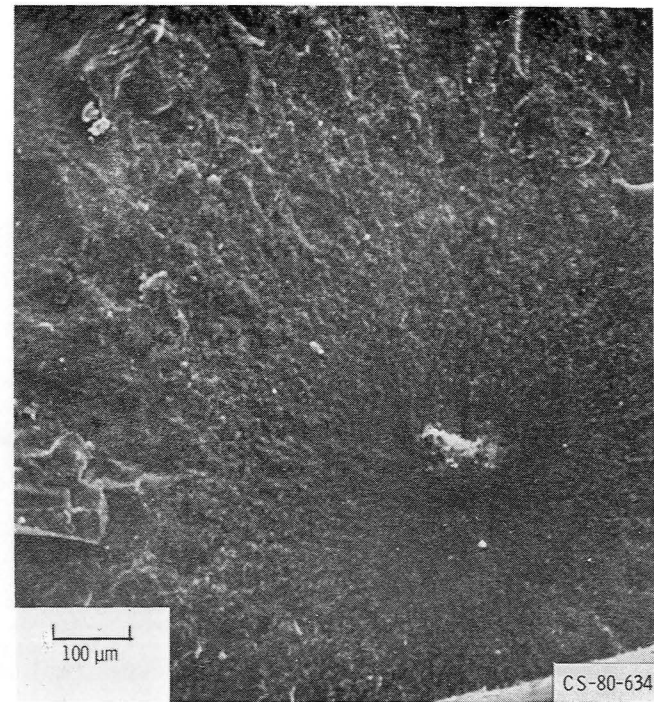


(b) NC-350.

Figure 6. - Room temperature fracture surfaces of RBSN.



(a) NC-350.



(b) BAR FROM WET ATTRITOR MILLED Si POWDER.

Figure 7. - Fracture initiating flaws in RBSN (room temperature fractures).

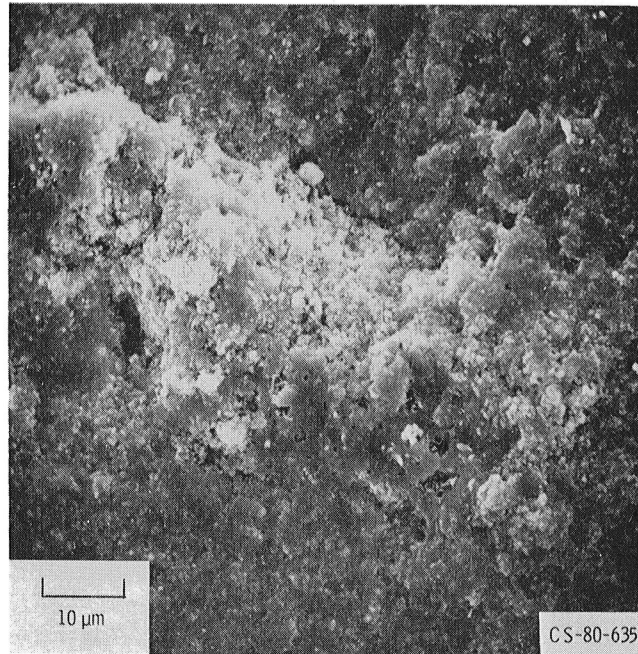
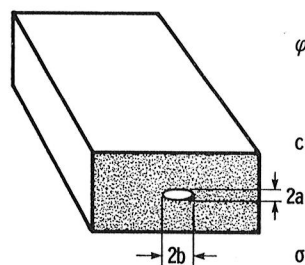


Figure 8. - Flaw showing spongy appearance in experimental RBSN fractured at room temperature.



$$c = \frac{\varphi^2 K_{IC}^2}{1.2 \pi \sigma^2}$$

$$\varphi = \int_0^{\pi/2} \left(\cos^2 \theta + \frac{a^2}{b^2} \sin^2 \theta \right)^{1/2} d\theta$$

c = CALCULATED FLAW SIZE CORRESPONDING TO
THE SMALLER OF a AND b

σ = OBSERVED FRACTURE STRESS

K_{IC} = CRITICAL STRESS INTENSITY FACTOR

Figure 9. - Calculation of predicted flaw size.

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